EXHIBIT 13

PURIFICATION OF LABORATORY CHEMICALS

Fourth Edition

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Preface to the Fourth Edition

THE AIMS of the first three editions, to provide purification procedures of commercially available chemicals and biochemicals from published literature data, are continued in this fourth edition. Since the third edition in 1988 the number of new chemicals and biochemicals which have been added to most chemical and biochemical catalogues have increased enormously. Accordingly there is a need to increase the number of entries with more recent useful reagents and chemical and biochemical intermediates. With this in mind, together with the need to reorganise and update general purification procedures, particularly in the area of biological macromolecules, as well as the time lapse since the previous publication, this fourth edition of **Purification of Laboratory Chemicals** has been produced. Chapter 1 has been reorganised with some updating, and by using a smaller font it was kept to a reasonable number of pages. Chapters 2 and 5 were similarly altered and have been combined into one chapter. Eight hundred and three hundred and fifty entries have been added to Chapters 3 (25% increase) and 4 (44% increase) respectively, and four hundred entries (310% increase) were added to Chapter 5 (Chapter 6 in the Third Edition), making a total of 5700 entries; all resulting in an increase from 391 to 529 pages, i.e. by *ca* 35%.

Many references to the original literature have been included remembering that some of the best references happened to be in the older literature. Every effort has been made to provide the best references but this may not have been achieved in all cases. Standard abbreviations, listed on page 1, have been used throughout this edition to optimise space, except where no space advantage was achieved, in which cases the complete words have been written down to improve the flow of the sentences.

With the increasing facilities for information exchange, chemical, biochemical and equipment suppliers are making their catalogue information available on the Internet , e.g. Aldrich-Fluka-Sigma catalogue information is available on the World Wide Web by using the address http://www.sigma.sial.com, and GIBCO BRL catalogue information from http://www.lifetech.com, as well as on CD-ROMS which are regularly updated. Facility for enquiring about, ordering and paying for items is available *via* the Internet. CAS on-line can be accessed on the Internet, and CAS data is available now on CD-ROM. Also biosafety bill boards can similarly be obtained by sending SUBSCRIBE SAFETY John Doe at the address "listserv@uvmvm.uvm.edu", SUSCRIBE BIOSAFETY at the address "listserv@mitvma.mit.edu", and SUBSCRIBE RADSAF at the address "listserv@romulus.ehs.uiuc.edu"; and the Occupational, Health and Safety information (Australia) is available at the address "http://www.worksafe.gov.au/~wsa1". Sigma-Aldrich provide Material Safety data sheets on CD-ROMs.

It is with much sadness that Dr Douglas D. Perrin was unable to participate in the preparation of the present edition due to illness. His contributions towards the previous editions have been substantial, and his drive and tenacity have been greatly missed.

The Third Edition was prepared on an IBM-PC and the previous IBM files were converted into Macintosh files. These have now been reformatted on a Macintosh LC575 computer and all further data to complete the Fourth Edition were added to these files. The text was printed with a Hewlett-Packard 4MV -600dpi Laser Jet printer which gives a clearer resolution.

I thank my wife Dr Pauline M. Armarego, also an organic chemist, for the arduous and painstaking task of entering the new data into the respective files, and for the numerous hours of proofreading as well as the corrections of typographic errors in the files. I should be grateful to my readers for any comments, suggestions, amendments and criticisms which could, perhaps, be inserted in the second printing of this edition.

W.L.F. Armarego
30 June 1996

Preface to the First Edition

WE BELIEVE that a need exists for a book to help the chemist or biochemist who wishes to purify the reagents she or he uses. This need is emphasised by the previous lack of any satisfactory central source of references dealing with individual substances. Such a lack must undoubtedly have been a great deterrent to many busy research workers who have been left to decide whether to purify at all, to improvise possible methods, or to take a chance on finding, somewhere in the chemical literature, methods used by some previous investigators.

Although commercially available laboratory chemicals are usually satisfactory, as supplied, for most purposes m scientific and technological work, it is also true that for many applications further purification is essential.

With this thought in mind, the present volume sets out, firstly, to tabulate methods, taken from the literature, for purifying some thousands of individual commercially available chemicals. To help in applying this information, two chapters describe the more common processes currently used for purification in chemical laboratories and give fuller details of new methods which appear likely to find increasing application for the same purpose. Finally, for dealing with substances not separately listed, a chapter is included setting out the usual methods for purifying specific classes of compounds.

To keep this book to a convenient size, and bearing in mind that its most likely users will be laboratory-trained, we have omitted manipulative details with which they can be assumed to be familiar, and also detailed theoretical discussion. Both are readily available elsewhere, for example in Vogel's very useful book **Practical Organic Chemistry** (Longmans, London, 3rd ed., 1956), or Fieser's **Experiments in Organic Chemistry** (Heath, Boston, 3rd ed, 1957).

For the same reason, only limited mention is made of the kinds of impurities likely to be present, and of the tests for detecting them. In many cases, this information can be obtained readily from existing monographs.

By its nature, the present treatment is not exhaustive, nor do we claim that any of the methods taken from the literature are the best possible. Nevertheless, we feel that the information contained in this book is likely to be helpful to a wide range of laboratory workers, including physical and inorganic chemists, research students, biochemists, and biologists. We hope that it will also be of use, although perhaps to only a limited extent, to experienced organic chemists.

We are grateful to Professor A. Albert and Dr D.J. Brown for helpful comments on the manuscript.

D.D.P., W.L.F.A. & D.R.P.
1966

Preface to the Second Edition

SINCE the publication of the first edition of this book there have been major advances in purification procedures. Sensitive methods have been developed for the detection and elimination of progessively lower levels of impurities. Increasingly stringent requirements for reagent purity have gone hand-in-hand with developments in semiconductor technology, in the preparation of special alloys and in the isolation of highly biologically active substances. The need to eliminate trace impurities at the micro- and nanogram levels has placed greater emphasis on ultra purification technique. To meet these demands the range of purities of laboratory chemicals has become correspondingly extended. Purification of individual chemicals thus depends more and more critically on the answers to two questions -Purification from what, and to what permissible level of contamination. Where these questions can be specifically answered, suitable methods of purification can usually be devised.

Several periodicals devoted to ultra purification and separations have been started. These include "Progress in Separation and Purification" Ed. (vol. I) E.S. Perry, Wiley-Interscience, New York, vols. 1-4, 1968-1971, and **Separation and Purification Methods** Ed. E S.Perry and C.J.van Oss, Marcel Dekker, New York, vol. 1-, 1973-. Nevertheless, there still remains a broad area in which a general improvement in the level of purity of many compounds can be achieved by applying more or less conventional procedures. The need for a convenient source of information on methods of purifying available laboratory chemicals was indicated by the continuing demand for copies of this book even though it had been out of print for several years.

We have sought to revise and update this volume, deleting sections that have become more familiar or less important, and incorporating more topical material. The number of compounds in Chapters 3 and 1 have been increased appreciably. Also,

We take this opportunity to thank users of the first edition who pointed out errors and omissions, or otherwise suggested improvements or additional material that should be included. We are indebted to Mrs S.Schenk who emerged from retirement to type this manuscript.

D.D.P., W.L.F.A. & D.R.P.
1980

Preface to the Third Edition

THE CONTINUING demand for this monograph and the publisher's request that we prepare a new edition, are an indication that **Purification of Laboratory Chemicals** fills a gap in many chemists' reference libraries and laboratory shelves. The present volume is an updated edition which contains significantly more detail than the previous editions, as well as an increase in the number of individual entries and a new chapter.

Additions have been made to Chapters 1 and 2 in order to include more recent developments in techniques (e.g. Schlenk-type, *cf* p. 10), and chromatographic methods and materials. Chapter 3 still remains the core of the book, and lists in alphabetical order relevant information on *ca* 4000 organic compounds. Chapter 4 gives a smaller listing of *ca* 750 inorganic and metal-organic substances, and makes a total increase of ca 13% of individual entries in these two chapters. Some additions have also been made to Chapter 5.

We are currently witnessing a major development in the use of physical methods for purifying large molecules and macromolecules, especially of biological origin. Considerable developments in molecular biology are apparent in techniques for the isolation and purification of key biochemicals and substances of high molecular weight. In many cases something approaching homogeneity has been achieved, as evidenced by electrophoresis, immunological and other independent criteria. We have consequently included a new section, Chapter 6, where we list upwards of 100 biological substances to illustrate their current methods of purification. In this chapter the details have been kept to a minimum, but the relevant references have been included.

The lists of individual entries in Chapters 3 and 4 range in length from single line entries to *ca* one page or more for solvents such as acetonitrile, benzene, ethanol and methanol. Some entries include information such as likely contaminants and storage conditions. More data referring to physical properties have been inserted for most entries [i.e. melting and boiling points, refractive indexes, densities, specific optical rotations (where applicable) and UV absorption data]. Inclusion of molecular weights should be useful when deciding on the quantities of reagents needed to carry out relevant synthetic reactions, or preparing analytical solutions. The Chemical Abstracts registry numbers have also been inserted for almost all entries, and should assist in the precise identification of the substances.

In the past ten years laboratory workers have become increasingly conscious of safety in the laboratory environment. We have therefore in three places in Chapter 1 (pp. 3 and 33, and bibliography p. 52) stressed more strongly the importance of safety in the laboratory. Also, where possible, in Chapters 3 and 4 we draw attention to the dangers involved with the manipulation of some hazardous substances.

The world wide facilities for retrieving chemical information provided by the Chemical Abstract Service (CAS on-line) have made it a relatively easy matter to obtain CAS registry numbers of substances, and most of the numbers in this monograph were obtained *via* CAS on-line. We should point out that two other available useful files are CSCHEM and CSCORP which provide, respectively, information on chemicals (and chemical products) and addresses and telephone numbers of the main branch offices of chemical suppliers.

The present edition has been produced on an IBM PC and a Laser Jet printer using the **Microsoft Word (4.0)** word-processing program with a set stylesheet. This has allowed the use of a variety of fonts and font sizes which has made the presentation more attractive than in the previous edition. Also, by altering the format and increasing slightly the sizes of the pages, the length of the monograph has been reduced from 568 to 391 pages. The reduction in the number of pages has been achieved in spite of the increase of *ca* 15% of total text.

We extend our gratitude to the readers whose suggestions have helped to improve the monograph, and to those who have told us of their experiences with some of the purifications stated in the previous editions, and in particular with the hazards that they have encountered. We are deeply indebted to Dr M.D. Fenn for the several hours that he has spent on the terminal to provide us with a large number of CAS registry numbers.

This monograph could not have been produced without the expert assistance of Mr David Clarke who has spent many hours to load the necessary fonts in the computer, and for advising one of the authors (W.L.F.A.) on how to use them together with the idiosyncrasies of Microsoft Word.

D.D.P. & W.L.F.A.
1988

CHAPTER 3

PURIFICATION OF ORGANIC CHEMICALS

The general principles, techniques and methods of purification in Chapters 1 and 2 are applicable in this chapter. Most organic liquids and a number of solids can readily be purified by fractional distillation, usually at atmospheric pressure. Sometimes, particularly with high boiling or sensitive liquids, or when in doubt about stability, distillation or fractionation under reduced pressure should be carried out. To save space, the present chapter omits many substances for which the published purification methods involve simple distillation. Where boiling points are given, purification by distillation is another means of removing impurities. Literature references are omitted for methods which require simple recrystallisation from solution if the correct solvent can be guessed readily, and where no further information is given, e.g. spectra. Substances are listed alphabetically, usually with some criteria of purity, giving brief details of how they can be purified. Also noted are the molecular weights (to the first decimal place), melting points and/or boiling points together with the respective densities and refractive indexes for liquids, and optical rotations when the compounds are chiral. When the temperatures and/or the wavelengths are not given for the last three named properties then they should be assumed to be 20°C and the average of the wavelengths of the sodium D lines repectively; and densities are relative to water at 4°.

The present chapter includes commercially available organic chemicals. Most of the organo- phosphorus, boron, silicon, alkali metal compounds and metal ion salts are in Chapter 4. Naturally occurring commercially available organic compounds of use in biochemistry, molecular biology and biology are included in Chapter 5.

Abbreviations of words and some journal names are listed in Chapter 1, pages 1 and 2.

As a good general rule all low boiling (<100°) organic liquids should be treated as highly flammable and the necessary precautions should be taken.

Abietic acid *[514-10-3]* **M 302.5, m 172-175°, $[\alpha]_D^{25}$ -116° (-106°)(c 1, EtOH).** Crystd by dissolving 100g of acid in 95% EtOH (700ml), adding to H_2O (600ml) and cooling. Filter, dry in a vacuum (over KOH or $CaSO_4$) store in an O_2-free atmosphere. λ in EtOH nm(log ε): 2343(4.3), 241(4.4), 2505(4.2), 235(4.34) and 240(4.36). [*Org Synth* **23** 1 *1952* ; *JACS* **35** 3736 *1949*; *M* **116** 1345 *1985*].

Abscisic acid *[21293-39-8]* **M 264.3, m 160-161° (sublimation), $[\alpha]_{287}$ + 24,000°, $[\alpha]_{245}$ -69,000° (c 1-50μg/ml in acidified MeOH or EtOH).** Crystd from CCl_4-pet.ether.

Acenaphthalene *[208-96-8]* **M 152.2, m 92-93°.** Dissolved in warm redistd MeOH, filtered through a sintered glass funnel and cooled to -78° to ppte the material as yellow plates [Dainton, Ivin and Walmsley *TFS* **56** 1784 *1960*]. Alternatively can be sublimed *in vacuo*.

Acenaphthaquinone *[82-86-0]* **M 182.2, m 260-261°.** Extracted with, then recrystd twice from C_6H_6. [LeFevre, Sundaram and Sundaram *JCS* 974 *1963*].

Acenaphthene *[83-32-9]* **M 154.2, m 94.0°.** Crystd from EtOH. Purified by chromatography from CCl_4 on alumina with benzene as eluent [McLaughlin and Zainal *JCS* 2485 *1960*].

2,3-Dimethylbenzoic acid *[603-79-21]* **M 150.2, m 146°.** Crystd from EtOH and is volatile in steam.

2,4-Dimethylbenzoic acid *[611-01-8]* **M 150.2, m 126-127°, b 267°/727mm.** Crystd from EtOH, and sublimed in a vacuum.

2,5-Dimethylbenzoic acid *[610-72-0]* **M 150.2, m 134°, b 268°/760mm,**
2,6-Dimethylbenzoic acid *[632-46-2]* **M 150.2, m 117°.** Steam distd, and crystd from EtOH.

3,4-Dimethylbenzoic acid *[619-04-5]* **M 150.2, m 166°.** Crystd from EtOH and sublimed *in vacuo*.

3,5-Dimethylbenzoic acid *[419-06-9]* **M 150.2, m 170°.** Distd in steam, crystd from water or EtOH and sublimed in a vacuum.

4,4'-Dimethylbenzophenone *[54323-31-8]* **M 210.3, m 95°, b 333-334°/725mm.** Purified by zone refining.

2,5-Dimethyl-1,4-benzoquinone *[137-18-8]* **M 136.1, m 124-125°.** Crystd from EtOH.

2,6-Dimethyl-1,4-benzoquinone *[527-61-7]* **M 136.1, m 72° (sealed tube).** Crystd from water/EtOH (8:1).

2,3-Dimethylbenzothiophene *[31317-17-6]* **M 212.3, b 123-124°/10mm, n^{19} 1.6171.** Fractionated through a 90cm Monel spiral column.

***N,N*-Dimethylbenzylamine** *[103-83-3]* **M 135.2, b 66-67°/15mm, 181°/760mm, d 0.900, n 1.501.** Refluxed with acetic anhydride for 24h, then fractionally distd. The middle fraction was dried with KOH, distd under reduced pressure, and stored under vacuum. Distn of the amine with zinc dust, at reduced pressure, under nitrogen, has also been used.

4,4'-Dimethyl-2,2'-bipyridine *[1134-35-6]* **M 184.2, m 175-176°.** Crystd from ethyl acetate. [Elliott et al. *JACS* **107** 4647 *1985*].

1,1'-Dimethyl-4,4'-bipyridylium dichloride ($3H_2O$; Methyl Viologen Dichloride, paraquat dichloride) *[1910-42-5]* **M 311.2, m >300°(dec).** Recrystd from MeOH/acetone mixture. Also crystd three times from absolute EtOH [Bancroft et al. *AC* **53** 1390 *1981*]. Dried at 80° in a vacuum.

***N,N*-Dimethylbiuret** *[7710-35-2]* **M 131.1.** Purified by repeated crystn from the melt.

2,3-Dimethyl-1,3-butadiene *[513-81-5]* **M 82.2, m -69-70°, b 68-69°/760mm, d 0.727, n 1.4385.** Distd from $NaBH_4$, and purified by zone melting.

1,3-Dimethylbutadiene sulphone *[10033-92-8]* **M 145.2, m 40.4-41.0°.** Crystd from ethyl ether.

2,2-Dimethylbutane *[75-83-2]* **M 86.2, b 49.7°, d 0.649, n^{25} 1.36595.** Distd azeotropically with MeOH, then washed with water, dried and distd.

2,3-Dimethylbutane *[79-29-8]* **M 86.2, b 58.0°, d 1.375, n^{25} 1.37231.** Distd from sodium, passed through a column of silica gel (activated by heating in nitrogen to 350° before use) to remove unsaturated impurities, and again distd from sodium. Also distilled azeotropically with MeOH, then washed with water, dried and redistd.

2,3-Dimethylbut-2-ene *[563-79-1]* **M 84.2, b 72-73°/760mm, d 0.708, n 1.41153.** Purified by GLC on a column of 20% squalene on chromosorb P at 50° [Flowers and Rabinovitch *JPC* **89** 563 *1985*]. Also washed with 1M NaOH soln followed by H_2O. Dried over Na_2SO_4, distd over powdered KOH under

nitrogen and passed through activated alumina before use. [Woon et al. *JACS* **108** 7990 *1986*; Wong et al. *JACS* **109** 3428 *1987*].

Dimethylcarbamoyl chloride *[79-44-7]* **M 107.5, m -33°, b 34°/0.1mm, d 1.172, n 1.4511.** Must distil under high vacuum to avoid decomposition.

3,3'-Dimethylcarbanilide *[620-50-8]* **M 240.3, m 225°.** Crystd from ethyl acetate.

Dimethyl carbonate *[616-38-5]* **M 90.1, m 4.65°, b 90-91°, d 1.070, n 1.369.** Contains small amounts of water and alcohol which form azeotropes. Stood for several days in contact with Linde type 4A molecular sieves, then fractionally distd. The middle fraction was frozen slowly at 2°, several times, retaining 80% of the solvent at each cycle.

***cis-and trans*-1,4-Dimethylcyclohexane** *[589-90-2]* **M 112.2, b 120°, d 0.788, n 1.427.** Freed from olefines by shaking with conc H_2SO_4, washing with water, drying and fractionally distilling.

5,5-Dimethyl-1,3-cyclohexanedione see **dimedone.**

1,2-Dimethylcyclohexene *[1674-10-8]* **M 110.2, b 135-136°/760mm, d 0.826, n 1.4591.** Passed through a column of basic alumina and distd.

1,5-Dimethyl-1,5-diazaundecamethylene polymethobromide (Hexadimethrene, polybrene) *[28728-55-4]*. Purified by chromatography on Dowex 50 and/or by filtration through alumina before use [Frank *Hoppe-Seyler's Z Physiol Chemie* **360** 997 *1979*].

Dimethyldihydroresorcinol see **dimedone**.

2,9-Dimethyl-4,7-diphenyl-1,10-phenanthroline *[4733-39-5]* **M 360.5, m >280°.** Purified by recrystn from benzene.

Dimethyl disulphide *[624-92-0]* **M 94.2, f.p. -98°, b 40°/12mm, 110°/760mm, d 1.0605, n 1.5260.** Passed through neutral alumina before use.

Dimethyl ether see **methyl ether.**

2,2-Dimethylethyleneimine *[2658-24-4]* **M 71.1, b 70.5-71.0°.** Freshly distd from sodium before use.

***N,N*-Dimethyl formamide (DMF)** *[68-12-2]* **M 73.1, b 76°/39mm, 153°/760mm, d 0.948, n^{25} 1.4269.** Decomposes slightly at its normal boiling point to give small amounts of dimethylamine and carbon monoxide. The decomposition is catalysed by acidic or basic materials, so that even at room temperature DMF is appreciably decomposed if allowed to stand for several hours with solid KOH, NaOH or CaH_2. If these reagents are used as dehydrating agents, therefore, they should not be refluxed with the DMF. Use of $CaSO_4$, $MgSO_4$, silica gel or Linde type 4A molecular sieves is preferable, followed by distn under reduced pressure. This procedure is adequate for most laboratory purposes. Larger amounts of water can be removed by azeotropic distn with benzene (10% v/v, previously dried over CaH_2), at atmospheric pressure: water and benzene distil below 80°. The liquid remaining in the distn flask is further dried by adding $MgSO_4$ (previously ignited overnight at 300-400°) to give 25g/L. After shaking for one day, a further quantity of $MgSO_4$ is added, and the DMF distd at 15-20mm pressure through a 3-ft vacuum-jacketed column packed with steel helices. However, $MgSO_4$ is an inefficient drying agent, leaving about 0.01M water in the final DMF. More efficient drying (to around 0.001-0.007M water) is achieved by standing with powdered BaO, followed by decanting before distn, with alumina powder (50g/L; previously heated overnight to 500-600°), and distilling from more of the alumina; or by refluxing at 120-140° for 24h with triphenylchlorosilane (5-10g/L), then distilling at *ca* 5mm pressure [Thomas and Rochow JACS 79 1843 1957]. Free amine in DMF can be detected by colour reaction with 1-fluoro-2,4-dinitrobenzene. It has also been purified by drying overnight over KOH pellets and then distd

from BaO through a 10 cm Vigreux column {*Experimental Cell Research* **100** 213 *1976*]. [For efficiency of desiccants in drying dimethyl formamide see Burfield and Smithers [*JOC* **43** 3966 *1978*, and for a review on purification, tests of purity and physical properties, see Juillard *PAC* **49** 885 *1977*].
It has been purified by distilling from K_2CO_3 under high vac and fractionated in an all-glass apparatus. The middle fraction is collected, degassed (seven or eight freeze-thaw cycles) and redistd under as high a vacuum as possible [Mohammad and Kosower *JACS* **93** 2713 *1971*].

***d,l*-2,4-Dimethylglutaric acid** *[2121-67-7]* **M 160.2, m 144-145°.** Distd in steam and crystd from ether/pet ether.

3,3-Dimethylglutaric acid *[4839-46-7]* **M 160.2, m 103-104°, b 89-90°/2mm, 126-127°/4.5mm.** Crystd from water, benzene or ether/pet ether. Dried in a vacuum.

3,3-Dimethylglutarimide *[1123-40-6]* **M 141.2, m 144-146°.** Recrystd from EtOH [Arnett and Harrelson *JACS* **109** 809 *1987*].

***N,N*-Dimethylglycinehydrazide hydrochloride** *[539-64-0]* **M 153.6, m 181°.** Crystd by adding EtOH to a conc aqueous soln.

Dimethylglyoxime *[95-45-4]* **M 116.1, m 240°.** Crystd from EtOH (10ml/g) or aqueous EtOH.

2,5-Dimethyl-2,4-hexadiene *[764-13-6]* **M 110.2, f.p. 14.5°, b 132-134°, d 0.773, n 1.4796.** Distd, then repeatedly fractionally crystd by partial freezing. Immediately before use, the material was passed through a column containing Woelm silica gel (activity I) and Woelm alumina (neutral) in separate layers.

2,2-Dimethylhexane *[590-73-8]* **M 114.2, m -121.2°, b 107°, d 0.695,**
2,5-Dimethylhexane *[592-13-2]* **M 114.2, m -91.2°, b 109°, d 0.694.** Dried over type 4A molecular sieves and distd.

2,5-Dimethylhexane-2,5-diol *[110-03-2]* **M 146.2, m 88-90°.** Purified by fractional crystn. Then the diol was dissolved in hot acetone, treated with activated charcoal, and filtered while hot. The soln was cooled and the diol was filtered off and washed well with cold acetone. The crystn process was repeated several times and the crystals were dried under a vac in a freeze-drying apparatus [Goates et al. *JCSFT 1* **78** 3045 *1982*].

5,5-Dimethylhydantoin *[77-71-4]* **M 128.1, m 177-178°.** Crystd from EtOH and sublimed *in vacuo.*

1,1-Dimethylhydrazine *[57-14-7]* **M 60.1, b 60.1°/702mm, d 0.790, n 1.408.** Fractionally distd through a 4-ft column packed with glass helices. Ppted as its oxalate from ethyl ether soln. After crystn from 95% EtOH, the salt was decomposed with aqueous saturated NaOH, and the free base was distd, dried over BaO and redistd [McBride and Kruse *JACS* **79** 572 *1957*]. Distn and storage should be under nitrogen.

4,6-Dimethyl-2-hydroxypyrimidine *[108-79-2]* **M 124.1, m 198-199°.** Crystd from absolute EtOH (charcoal).

1,2-Dimethylimidazole *[1739-84-0]* **M 96.1, b 206°/760mm, d 1.084.** Crystd from benzene and stored at 0-4°. [Gorun et al. *JACS* **109** 4244 *1987*].

1,1-Dimethylindene *[18636-55-0]* **M 144.2.** Purified by gas chromatography.

Dimethyl itaconate *[617-52-7]* **M 158.2, m 38°, b 208°, d 1.124.** Crystd from MeOH by cooling to -78°.

Dimethylmaleic anhydride *[766-39-2]* **M 126.1, m 96°, b 225°/760mm.** Distd from benzene/ligroin and sublimed in a vacuum.